

AD-A193 467

THE RADIATION SENSITIVITY OF SELECT METAL CHELATE  
POLYMERS: MECHANISTIC C. C. (U) MASSACHUSETTS UNIV AMHERST  
DEPT OF CHEMISTRY R D ARCHER ET AL. 01 OCT 87 18-11  
AD0014-86-R-0345

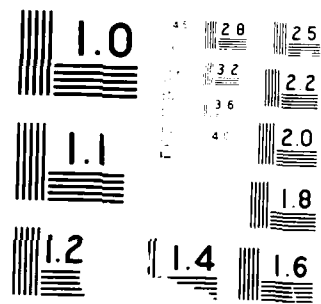
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REPORT DOCUMENTATION PAGE			
1a REPORT SECURITY CLASSIFICATION <b>AD-A193 467</b>		1b RESTRICTIVE MARKINGS <b>DTIC</b>	
2 DATE 11 1988		3 DISTRIBUTION AVAILABILITY OF REPORT Approved for public release. Distribution unlimited.	
4 AUTHOR Technical Report Number 11		5 MONITORING ORGANIZATION REPORT NUMBER(S) Office of Naval Research	
6a NAME OF PERFORMING ORGANIZATION University of Massachusetts	6b OFFICE SYMBOL (If applicable)	7a NAME OF MONITORING ORGANIZATION Office of Naval Research	
6c ADDRESS (City, State, and ZIP Code) Dept. of Chemistry Univ. of Massachusetts Amherst, MA 01003		7b ADDRESS (City, State, and ZIP Code) 800 N. Quincy Street Arlington, VA 22217	
8a NAME OF FUNDING SPONSORING ORGANIZATION Office of Naval Research	8b OFFICE SYMBOL (If applicable)	9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER N00014-86-K-0345	
8c ADDRESS (City, State and ZIP Code) 800 N. Quincy St. Arlington, VA 22217		10 SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO	10 SOURCE OF FUNDING NUMBERS PROJECT NO
		10 SOURCE OF FUNDING NUMBERS TASK NO	10 SOURCE OF FUNDING NUMBERS WORK UNIT ACCESSION NO
11 TITLE (Include Security Classification) The Radiation Sensitivity of Select Metal Chelate Polymers: Mechanistic Changes at Higher Energies (Unclassified)			
12 PERSONAL AUTHOR(S) Archer, Ronald Dean; Hardiman, Christopher John; and Lee, Annabel Y.			
13a TYPE OF REPORT Publication	13b TIME COVERED FROM TO	14 DATE OF REPORT (Year, Month, Day) October 1, 1987	15 PAGE COUNT 11
16 SUPPLEMENTARY NOTATION Publication to appear in Photochemistry and Photophysics of Coordination Compounds, edited by H. Yersin & A. Vogler, Springer-Verlag, Berlin			
17 COSATI CODES FIELD GROUP SUB-GROUP		18 SUBJECT TERMS (Continue on reverse if necessary and identify by block number) radiation sensitive polymers metal coordination polymers uranyl polymers dioxouranium(VI) polymers positive resists negative resists	
19 ABSTRACT (Continue on reverse if necessary and identify by block number) Based on uranyl photochemistry in the visible/ultraviolet region, uranyl carboxylates are known to undergo CO <sub>2</sub> evolution; however, with gamma radiation (cesium-137, 662 keV) a sizable number of uranyldicarboxylate polymers crosslink with high efficiency and without CO <sub>2</sub> evolution. On the other hand several dicarboxylates with sulfur in the organic backbone exhibit very high scission efficiency. G values in excess of normal organic resists is related to the heavy atom content of these metal coordination polymers.			
20 DISTRIBUTION AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21 ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a NAME OF RESPONSIBLE INDIVIDUAL Dr. Kenneth Wynne		22b TELEPHONE (Include Area Code) (202) 9696-4409	22c OFFICE SYMBOL

OFFICE OF NAVAL RESEARCH  
Contract No. N00014-86-K-0345  
TECHNICAL REPORT NO. 11

THE RADIATION SENSITIVITY OF SELECT METAL CHELATE POLYMERS:  
MECHANISTIC CHANGES AT HIGHER ENERGIES

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Accepted for Publication  
in

Photochemistry and Photophysics of Coordination Compounds  
H. Yersin and A. Vogler, editors  
Springer-Verlag, Berlin, Publishers

October 1, 1987

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INTRODUCTION

Our interest in the radiation sensitivity of heavy metal polymers was first sparked by the goal of enhanced miniaturization of integrated circuit (IC) chip features. As the features of IC chips get closer and closer together at the submicron level, microlithographic techniques must use higher energy photons (or electrons, etc.) than are currently used.<sup>1</sup> Otherwise, diffraction effects by the photons which pass through adjacent features blur the features and allow short circuits. The thin organic polymer films, which are normally used as lithographic resists, suffer from an inability to absorb all of the higher energy radiation. The unabsorbed radiation backscatters from the oxide layer which is normally between the resist and the semiconductor.

Heavy atom-containing polymer films should have an enhanced sensitivity to applied radiation because of the atomic number ( $Z$ ) dependence of the photoelectric effect which has an atomic absorption coefficient which is  $Z^4$  or greater (Evans 1972) whereas photon scattering by heavy atoms only has a  $Z^2$  dependence. To test this concept,<sup>2</sup> we synthesized and tested a number of uranyl dicarboxylate polymers for gamma-ray

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1 Visible and ultraviolet radiation passed through masks or projected onto the surface provide the mass production necessary for the low cost devices currently on the market. Although electron beam lithography can provide higher resolution, the throughput rate and backscattering problems suggest that x-ray or low-energy gamma-ray methodology is needed. For more details see either of two recent review volumes (Thompson 1983, 1984)

2 Polymers with main group heavy elements have shown increased sensitivity using the empirical method of determining solubilities before and after radiation (Webb 1979; Haller 1979). Such sensitivities are based on the dose per unit area required to dissolve the irradiated polymer with 1/2 of the unirradiated polymer still on the substrate (for a positive resist and vice versa for a negative resist). They are solvent and laboratory dependent, cf. Thompson (1983, 1984), and are not as fundamental as  $G$  values.

sensitivity. From the extensive literature on uranyl carboxylate photochemistry, cf. Burrows (1974), we anticipated  $\text{CO}_2$  evolution, possibly mixed with hydrogen abstraction (Rehorek 1982). Much to our surprise, a sizable number of the uranyl polymers show no  $\text{CO}_2$  loss during  $^{137}\text{Cs}$  irradiation, but they crosslink with very high efficiency instead.

#### METHODOLOGY

The irradiation studies were conducted with a cesium-137 gamma photon source (662 keV) with dose rates of 0.022 to 0.073 Mrad/hr based on Fricke dosimetry (Hine 1956; Getoff 1962). Corrections for different mass absorption cross sections between the dosimeter and the uranyl polymers were made subsequently using literature data (Mann 1960) interpolated when necessary. Samples were irradiated in vacuo using sealed Pyrex or quartz tubes. Molecular weight analysis of the polymers before and after irradiation were conducted via gel permeation (or size exclusion) chromatography (GPC) in N-methylpyrrolidone (NMP) solutions on Ultrastyrigel columns, which had been calibrated with polystyrene standards (in NMP). Gas chromatography-mass spectroscopy (GCMS), electron spin resonance (ESR), Fourier transform nuclear magnetic resonance (NMR) and infrared (IR), and ultraviolet-visible (UVV) spectroscopies were used to help elucidate the products.

G values were determined using the method of Charlesby (1954, 1960, 1964) and reiterated by Dole (1973):

$$1/\bar{M}_n' = 1/\bar{M}_n^0 + (G_s - G_x)(r/100N_a) \quad [1]$$

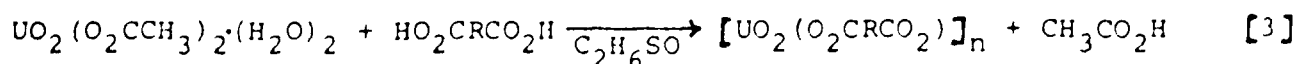
where  $\bar{M}_n^0$  is the number-average molecular weight before irradiation,  
 $\bar{M}_n'$  is the number-average molecular weight after irradiation,  
 $G_s$  is the number of chemical scissions per 100 electron volts,  
 $G_x$  is the number of crosslinks per 100 electron volts,  
 $r$  is the radiation dose in electron volts per gram, and  
 $N_a$  is Avogadro's number.

Converted to Megarads (Mrad) the equation becomes:

$$1/\bar{M}_n' = 1/\bar{M}_n^0 + (G_s - G_x)(1.04 \times 10^{-6} r) \quad [2]$$

These equations are based on random scission and crosslinking processes, random initial molecular weight distributions ( $\bar{M}_w/\bar{M}_n = 2$ ), and with  $G_s$  and  $G_x$  dose independent. Even for non-ideal systems, they provide the best guide possible to the magnitude of the radiation effects, and the dose dependence is apparent from plots of  $1/\bar{M}_n$  vs dose in Mrad.

The synthesis and characterization of the uranyl dicarboxylates is detailed elsewhere (Archer 1987; Hardiman 1987). The essential synthetic reaction is



The molecular weights have been determined by GPC, viscosity, and NMR end-group analysis. Solid-state  $^{13}\text{C}$ -NMR has also been used (Archer 1987) to confirm the coordination modes of the carboxylate ligands to the uranyl ion. The results are as anticipated from IR results (Hardiman 1987); that is, both monodentate and bidentate carboxylate coordination occur such that 7-coordination predominates in polymers with either one or two moles of solvent coordinated to the uranyl ion.

#### URANYL POLYMERS WHICH APPEAR TO EXCLUSIVELY CROSSLINK

As anticipated, the uranyl dicarboxylate polymers are very sensitive to gamma-ray irradiation. The data for four branched alkyl, one alkene, and one aromatic dicarboxylates which appear to exclusively crosslink, with no evidence of  $\text{CO}_2$  evolution, are summarized in Table 1. Whereas the entire absorption for the lighter elements (0.257, 1.54, 2.04, and 4.09 barns/atom for H, C, O, and S, respectively) is of the Compton type, for uranium 50% is photoelectric absorption (47.7 barns/atom total). Thus, the uranium atom constitutes from 50 to 58% of the absorption in the unit at this energy (662 keV), but only modifies the mass absorption coefficient for the polymer by about 13 to 16% relative to water, the absorber of the Fricke dosimeter. Organic polymers are typically lower than water; e.g., poly(methyl methacrylate), PMMA, is  $0.083 \text{ cm}^2/\text{g}$  using the same numbers, or 17 to 20% lower than the uranyl polymers. However, on a repeating unit basis, the uranyl polymers absorb from 600 to 700% as much radiation as PMMA ( $G = 1.3$ ) and organic polymers with  $G$  values as high as 10 are known (Thompson 1934).

None of the species in Table 1 show any tendency for scission, thus, the  $G_s - G_x$  value is essentially  $-G_x$ . With increased dose levels, all exhibit lower  $G_x$  values--a logical consequence of fewer effective crosslinks. Extra crosslinks between the same chains do not increase the molecular weight any further. The values in the table are for 2 Mrad doses (or 3 Mrad for the succinate and phthalate derivatives as no curvature was observed until above 3 Mrad for these two species). The extremely high  $G_x$  value for the tetrapimelate derivative must be related to the mobility of the longer alkyl chain coupled with stabilized tertiary radicals coupled with efficient energy transfer.

Table 1. Gamma-ray Sensitivity of Negative Resist Uranyl Polymers

Empirical Formula Unit <sup>a</sup> (bridging carboxylate)	$\bar{M}_n$ <sup>b</sup>	$G_s - G_x$ <sup>c</sup>	$\mu/\rho$ <sup>d</sup>	$G_s - G_x$ <sup>e</sup>
UO <sub>2</sub> O <sub>2</sub> CC(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> CO <sub>2</sub> (C <sub>2</sub> H <sub>6</sub> SO) (2,2-dimethylsuccinate)	9,000 <sup>g</sup>	-14 <sup>g</sup>	0.100	-12 <sup>g</sup>
UO <sub>2</sub> O <sub>2</sub> CC(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CO <sub>2</sub> (C <sub>2</sub> H <sub>6</sub> SO) (2,2-dimethylglutarate)	48,000 <sup>f</sup>	-3.1 <sup>f</sup>	0.100	-2.7 <sup>f</sup>
UO <sub>2</sub> O <sub>2</sub> CCH <sub>2</sub> C(CH <sub>3</sub> ) <sub>2</sub> CH <sub>2</sub> CO <sub>2</sub> (C <sub>2</sub> H <sub>6</sub> SO) (3,3-dimethylglutarate)	12,000 <sup>g</sup>	-8.0 <sup>g</sup>	0.100	-6.9 <sup>g</sup>
UO <sub>2</sub> O <sub>2</sub> CC(CH <sub>3</sub> ) <sub>2</sub> (CH <sub>2</sub> ) <sub>3</sub> C(CH <sub>3</sub> ) <sub>2</sub> CO <sub>2</sub> (C <sub>2</sub> H <sub>6</sub> SO) (2,2,6,6-tetramethylpimelate)	12,000 <sup>g</sup>	-43 <sup>g</sup>	0.099	-37 <sup>g</sup>
UO <sub>2</sub> (Z-O <sub>2</sub> CH=CHCO <sub>2</sub> ) (C <sub>2</sub> H <sub>6</sub> SO) <sub>1.75</sub> (maleate)	9,300	-3.3	0.099	-2.9
UO <sub>2</sub> (O-O <sub>2</sub> CC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> ) (C <sub>2</sub> H <sub>6</sub> SO) <sub>2</sub> (phthalate)	17,800 <sup>g</sup>	-3.7 <sup>g</sup>	0.097	-3.3 <sup>g</sup>

<sup>a</sup>Repeating unit of polymer chain including solvation

<sup>b</sup> $\bar{M}_n$  of samples not irradiated; based on GPC in NMP with polystyrene standards, or by NMR end-group analysis if so indicated

<sup>c</sup>Measured net G value using equation 2 and GPC after <sup>137</sup>Cs irradiation

<sup>d</sup>Mass absorption coefficients in cm<sup>2</sup>/g for 662 keV irradiation

<sup>e</sup>Corrected G values relative to Fricke dosimeter (0.086 cm<sup>2</sup>/g for <sup>137</sup>Cs)

<sup>f</sup>Polymer has poor  $\bar{M}$  distribution and poor agreement with end-group data

<sup>g</sup> $\bar{M}$  values for this polymer--end-group calibrated by NMR

#### URANYL POLYMERS WHICH UNDERGO SCISSION WITH GAMMA RADIATION

On the other hand, uranyl polymers with three different thio-bridged ligands and fumarate all show large  $G_s$  values upon irradiation as noted in Table 2. The cleanest one is the polymeric uranyl thiobis(succinate), which shows no tendency toward crosslinking; that is,  $G_s - G_x$  is probably a true measure of  $G_s$ . The GPC's of the others show evidence for both scission and crosslinking; thus  $G_s$  is larger than the  $G_s - G_x$  value shown. Unfortunately, they are so sensitive to radiation that reliable independent values are impossible to obtain. These high  $G_s - G_x$  values for the thio-bridged polymers would be even higher if the polystyrene-equivalent molecular weights were used instead of the NMR



Table 2. Gamma-ray Sensitivity of Positive Resist Uranyl Polymers<sup>f</sup>

Empirical Formula Unit <sup>a</sup> (bridging carboxylate)	$\bar{M}_n$ <sup>b</sup>	$G_S - G_X$ <sup>c</sup>	$\mu/\rho$ <sup>d</sup>	$G_S - G_X$ <sup>e</sup>
$UO_2(O_2CCH_2SCH_2CO_2)(C_2H_6SO)_2$ (thiodiglycolate)	14,000 <sup>g</sup>	56 <sup>g</sup>	0.097	50 <sup>g</sup>
$UO_2(O_2CCH_2SSCH_2CO_2)(C_2H_6SO)_{1.5}$ (dithiodiglycolate)	12,000 <sup>g-j</sup>	300 <sup>g-j</sup>	0.097	280 <sup>g-j</sup>
$UO_2(O_2CCH_2SCH_2SCH_2CO_2)(C_2H_6SO)_2$ (methylenebis thioglycolate)	15,000 <sup>g,h</sup>	103 <sup>g,h</sup>	0.096	93 <sup>g,h</sup>
$UO_2(E-O_2CCH=CHCO_2)(C_2H_6SO)_2$ (fumarate)	26,000 <sup>h,i</sup>	63 <sup>h,i</sup>	0.098	55 <sup>h,i</sup>

a-e Same as Table 1

f G values based on first 0.5 to 0.6 Mrad only

g M adjusted to agree with NMR end-group analysis

h Polymer undergoes both scission and crosslinking

i  $\bar{M}$  distribution poor; results very approximate

j Polymer very sensitive to radiation--nonirradiated samples change  $\bar{M}$  distribution significantly during irradiation of other samples

end-group determined weights. Specifically, thiodiglycolate, dithiodiglycolate, and methylenebis thioglycolate were originally calculated to have  $G_S - G_X$  values of about 100, 400, and 160, respectively, using the polystyrene-equivalent calibrations. These polymers absorb from about 5 to 7 times as much energy per polymer unit as an organic polymer (such as PMMA) with a molecular weight of 100 at this energy. Even so, with the ligands that have two sulfur atoms per repeating unit, extremely high sensitivity to this gamma radiation occurs. At low dose level, where PMMA shows only a very small change in molecular weight distribution, these uranyl species have drastically modified molecular weight distribution. GMS results indicate S-C scission for methylenebis thioglycolate and S-S scission in dithiodiglycolate. And ESR results indicate appreciable spin density on sulfur atoms for the same polymers with no evidence for uranium(V), even at -77°K.

The above results with uranyl polymers led us to synthesize linear cobalt(III) polymers containing two  $\beta$ -diketones bridged with S atoms (-S-, -SS-, -SSS-, etc.) of thiolates and ketones as well as thioamides and

bidentate ligand, which allows the synthesis of linear coordination polymers. These and similar  $\text{VO}^{2+}$   $\beta$ -diketonates bridged with sulfur atoms (Archer 1986) show radiation effects which are both wavelength and state/solvent dependent. For example, instead of reduction to cobalt(II), gamma irradiation produces facile C-S scission in the cobalt(III) polymers. Space limitations precludes further details.

#### ACKNOWLEDGEMENTS

The financial support of the U.S. Office of Naval Research and helpful interactions with Professors J.C.W. Chien (Univ. Massachusetts) and N. Getoff (Univ. Wien) are gratefully acknowledged.

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